

Mobile Laboratory Support to Site Mitigation: Simplified Method for Measuring Toxaphene Contamination by Gas Chromatography/Mass Spectrometry with Selective Ion Monitoring

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Mobile Laboratory Support to Site Mitigation: Simplified Method for Measuring Toxaphene Contamination by Gas Chromatography/Mass Spectrometry with Selective Ion Monitoring

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EXECUTIVE SUMMARY

Although the primary responsibility of the DTSC Mobile Laboratory (ML) is to rapidly respond to any emergency in California, the ML also supports Site Mitigation projects on request. During January 22–25, 2007, the ML team was deployed to Dos Palos, CA to help in the investigation of a site (Central Valley Fertilizer) contaminated with toxaphene. A simplified screening method was developed to identify and measure toxaphene with a Limit of Quantitation (LOQ) of 0.5 ppm. Using this method, the ML team successfully completed the analysis of 36 soil samples in less than 3 days. Toxaphene was measured in 6 of those samples above the action limit (1.8 ppm) set for this site. These on-site measurements provided project managers with timely information. An extraction step will be added to this method in the near future and a comparison against standard methods will be undertaken.

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ABBREVATIONS AND ACRONYMS

AMDIS = Automatic Mass Spectrum Deconvolution Identification Software GC/ECD = Gas Chromatograph/electron capture detector GC/MS = Gas Chromatograph/Mass Spectrometer GC/MSD = Gas Chromatograph/Mass Selective Detector NIMS = Negative Ion Mass Spectrometry SIM = Selective Ion Monitoring SOP = Standard Operating Procedure TIC = Total Ion Chromatogram

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INTRODUCTION

The Department of Toxic Substances Control's (DTSC) Mobile Laboratory (ML) was funded by the Homeland Security Grant Program (HSGP). The primary duty of the ML team is emergency response. Because of its mobile deployment capabilities, the ML team is designated to quickly identify unknown chemical releases from natural disasters, industrial spills, explosions or terrorist attacks.

Site Mitigation Projects are a major part of DTSC's responsibilities. Ongoing projects are essential for the protection of public health and the environment within the state. One of the difficulties for most project managers is obtaining timely assessment information about the contaminated site and/or to locate on-site hot spots. The ML team can be instrumental in Site Mitigation projects (1). A core capability of the ML is to characterize site contamination with qualitative and quantitative chemical data in order to quickly supply project managers with critical information for on-site decision making and risk assessment. In response to a request from the Site Mitigation Branch Project Manager, the ML team was deployed to Dos Palos, County of Merced, California, on January 22-25, 2007.

METHODS

Site Description

The abandoned property belongs to the Central Valley Fertilizer Company and consists of a cement bed for washing pesticide spraying equipment and vehicles, a waste water collection basin and a half acre evaporation pond. The sample plan and abandoned site are presented in Figure 1. The scope of the assignment was to characterize the site for toxaphene contamination with the objective of procuring risk assessment information for future cleanup or litigation.

Approach

A simplified method to analyze soil samples for toxaphene was developed for this Site Mitigation project. The method utilized the on-board GC/MS with a SIM mode to quickly screen soil samples for toxaphene. The project manager required a limit-of-quantitation (LOQ) level of 1.8 ppm; therefore, the limit-of-detection (LOD) level was set at 3 times below that, i.e., at 0.5 ppm.

Materials and Reagents

- Solvents Pesticide grade (Thermo Fisher Scientific, Pittsburgh, PA).
- 2. Standard Toxaphene, National Institute of Standards & Technology, Gaithersburg, MD 20899.
- 3. Internal Standard: 2-florobiphenyl (Supelco, 595 North Harrison Road, Bellefonte, PA).
- 4. Surrogate: decachlorobiphenyl (Supelco, 595 North Harrison Road, Bellefonte, PA).

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Apparatus

The ML consists of one vehicle (Ford F-555), a satellite communication system and two gas chromatograph/mass spectrometers (GC/MS) specifically designed for detecting toxic industrial chemicals in air, soil and water. The advanced analytical instruments are complemented by a series of field instruments for the quick detection of volatile compounds, metals and other chemicals. The GC/MS conditions used in this study were:

- 1. GC/MSD: Agilent 6850/5973 GC/MSD with ChemStation computer system.
- 2. Column: HP-5MS (5% phenyl methyl siloxane).
- 3. Temperature program: rate 20°C/min, initial temperature 80°C, final temperature 300°C and run time 14.5 min.
- 4. MSD/SIM setup: auto tune, DFTPP tune, toxaphene target ion 159 m/z, reference ions 231 m/z and 233 m/z..
- 5. Internal standard: 2-florobiphenyl target ion 172 m/z, reference ions 170 m/z and 171 m/z.
- 6. Surrogate: decachlorobiphenyl target ion 241 m/z, reference ions 170 m/z and 172 m/z.

Sample and Standard Preparation

Extraction: Approximately 5g soil were weighed into a 40 mL VOA vial, and 5mL of a hexane solution containing 2-fluorobipheny (1ug/mL) and decachlorobiphenyl (1ug/mL) as internal standard and surrogate, respectively, were added. The vial was extracted for approximately 3 min on a vortex mixer and approximately 1 mL of the top layer was taken into an autosampler vial for GC/MSD injection. If the soil samples were wet, anhydrous Na₂SO₄ (ca. 1.0g) was premixed before extraction.

Standards: Six level standards (0.5, 1.0, 2.0, 4.0, 8.0 and 10 ug/mL) were made from soil matrix blanks.

<u>Quantitation retention time</u>: the average of four retention time peaks (9.57, 9.91, 10.65 and 11.15 min) was used.

RESULTS AND DISCUSSION

Gas chromatogram peak retention time and peak area, as well as mass spectrum and ion abundance have been successfully used for volatile and semi-volatile organic compound identification and quantitation. But multi-component analytes, such as toxaphene, aroclors and chlordane present unusual difficulties due to their undefined peaks, multi-humps, and weathering effect changes. As such, these compounds present an interesting challenge. Furthermore, Method 8270C (2) has suggested that the recommended method to test these analytes is Method 8081A (3), i.e., using Gas Chromatography/electron capture detector (GC/ECD), rather than GC/MSD because of sensitivity limitations. A US Office of Inspector General Ombudsman Report recommended using chemical ionization technique to monitor toxaphene degradation products by negative ion mass spectrometry (NIMS) (4). However the ML's on-board GC/MSDs are standard-issue instruments for homeland security rapid identification; and a GC/MSD with Automatic Mass Spectrum Deconvolution Identification Software (AMDIS) superbly detects chemical agents at trace levels. Furthermore, space

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limitations make it infeasible to accommodate additional instruments such as a GC/ECD and GC/NIMS with their on-board computer systems. With these considerations in mind, a simplified method to analyze toxaphene by GC/SIM for site characterization was developed to respond to the site mitigation project.

Literature review (5) demonstrated successful results by averaging 4 predetermined retention time area counts for quantitation, therefore, 9.57, 9.91, 10.65 and 11.15 min were chosen after toxaphene hump shape pattern evaluation in the total ion chromatogram (TIC). To establish the quantitation table, 6 levels (0.5, 1.0, 2.0, 4.0, 8.0 and 10 ug/mL) standards were used for each retention time. All standards were made from a hexane extraction in pre-tested soil blanks to suppress soil matrix interference, increase the S/N ratio and enhance sensitivity. The correlation coefficient of the 6 level standards for 4 retention times were r²= 0.999 at 9.57 min, 0.997 at 9.91 min, 0.999 at 10.65 min and 0.999 at 11.15 min. Internal standard, 2-fluorobiphenyl, was used to obtain relative response factors in quantitation and surrogate decachlorobiphenyl was added into each sample for method recovery data.

The ML staff received 36 samples from the on-site project manager which were prepared according to the SOP (6). The samples were placed on the autosampler injector for an overnight run and the data were reviewed and processed the following day. The non-detected (ND) samples were screened out by visual comparison or by overlaying the extracted ion (159 m/z) chromatogram to the chromatogram of standards, the extracted ion chromatogram of which is presented in Figure 2. Because of the unique shape of toxaphene, this is a very effective way to differentiate negative samples from positive samples. One ND sample and the 8.0 ppm toxaphene positive sample are presented in Figures 3 and 4, respectively. The results of the positive samples were calculated based on the average of 4 retention time reported values, and the results are presented in Table 1 and Table2. The average surrogate (decachlorobiphenyl) recovery %, SD and CV % were 92.3, 12.2 and 13.2 from 36 samples respectively.

Development plans for a simple extraction and cleanup procedure are being considered for the near future, and a comparison against standard methods will be undertaken.

CONCLUSIONS

Site Mitigation is a major part of DTSC's responsibility. The ML scientists used their simplified GC/MS/SIM method to complete 36 samples in less than 3 days and to detect 6 contaminated hot spots on the site. This simplified method was developed for quick on-site screening and site characterization purposes. This method is simple, fast and easy to use as no lengthy extraction and cleanup procedures are necessary. However, this method may be biased towards false positives rather than false negatives; consequently, development plans for simple extraction and cleanup procedures are being considered for the near future.

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REFERENCES

- (1) DTSC, SOP 1000-S, Mobile Laboratory Activation and Deployment (2007)
- (2) EPA Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). Test methods for Evaluating Solid Waste, Physical/Chemical methods (SW-846).
- (3) EPA method 8081A, Organochlorine Pesticides by Gas Chromatography, Test Methods for Evaluating Solid Waste, Physical/Chemical methods (SW-846).
- (4) US Office of Inspector General Ombudsman Report (No. 2006-P- 00007). More Information Is Needed On Toxaphene Degradation Products, December 16, 2005.
- (5) PerkinElmer Instruments, Environmental Application Note.
- (6) DTSC, SOP 1018-S, Screening Method for Toxaphene in Soil (2007)
- (7) Food, Testing & Analysis 6, 13-18, 46. 2000

ACKNOWLEDGMENTS

The authors thank Maria Gillette, Project Manager and her associates, Brad Parsons, Jerry Lile and Sam Martinez, for preparing on-site homogenous soil samples. The authors also wish to thank Dr. Bruce La Belle (Division Chief) and Dr. Myrto Petreas (Branch Chief) for their support and encouragement of this research project.

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Table 1. Quantitative Results by Averaging Calibrated Results from 4 retention times

```
Quantitation Report
                                                        (QT Reviewed)
  Data Path : C:\msdchem\1\DATA\LOSBAN\
  Data File : STD070123A.D
  Acq On : 23 Jan 2007 12:25
Operator : OG /KC
  Sample : STD070123A
Misc : CHECK CALIB STD 4PPM
ALS Vial : 12 Sample Multiplier: 1
  Quant Time: Jan 23 13:44:43 2007
  Quant Method : C:\msdchem\1\METHODS\TXPN3LB.M
  Quant Title : TOXAPHENE 3 IONS SIM GCMS
  QLast Update : Tue Jan 23 10:53:28 2007
  Response via : Initial Calibration
                                             R.T. QIon Response Conc Units Dev(Min)
   Internal Standards
     1) Internal Std(2-FBP) R... 5.065 172 1277116
                                                                     1.00 ppm
     6) Surrogate RT 12.72 12.717 214
Spiked Amount 1.000
   System Monitoring Compounds
                                                          72864 1.11 ppm
Recovery = 111.00%
                                                                                         0.01
   Target Compounds
                                                                                      Qvalue
     2) Toxaphene RT9.57 9.572 159 1554m 4.61 ppm 3) Toxaphene RT9.91 9.910 159 2116 4.07 ppm 4) Toxaphene RT10.65 10.652 159 4481 4.18 ppm 5) Toxaphene RT11.15 11.157 159 3363m 4.65 ppm
                                                                                            90
                                                                                            83
   (\#) = qualifier out of range (m) = manual integration (+) = signals summed
                                                                           Average = 4.38 \text{ ppm}
TXPN3LB.M Tue Jul 24 10:05:22 2007
```

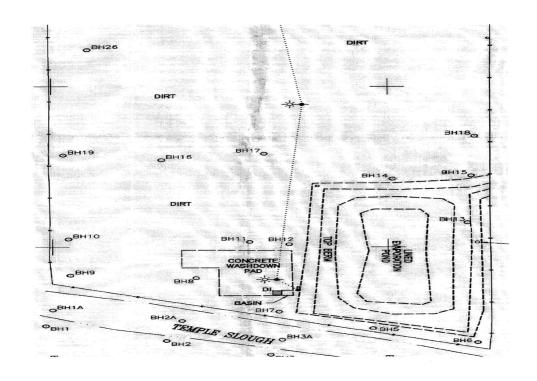
Table 2. Results of Toxaphene and Surrogate Recovery in soil samples

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ECL Number	Collector's Number	Toxaphene, mg/Kg	Decachlorobiphenyl, %
AQ01031	CVF P-1	< 1.8	81.0
AQ01031	CVF P-2	8.0	81.0
AQ01032	CVF P-3	6.8	82.0
AQ01033	BH-10-6-9	< 1.8	76.0
AQ01035	CVF-13-6-9	< 1.8	79.0
AQ01036	POND 1-24	< 1.8	85.0
AQ01037	POND 2-12	< 1.8	87.0
AQ01037	POND 2-2	< 1.8	86.0
AQ01039	POND 2-S	< 1.8	88.0
AQ01033 AQ01040	POND 3-24	< 1.8	88.0
AQ01040 AQ01041	POND 4-5	< 1.8	88.0
AQ01041 AQ01042	POND 4-12	< 1.8	80.0
AQ01042 AQ01043	POND 4-12	< 1.8	77.0
AQ01043 AQ01044	POND 5-S	< 1.8	87.0
AQ01044 AQ01045	CVF POND 6-1	< 1.8	78.0
AQ01045 AQ01046	CVF POND6-2	< 1.8	78.0
AQ01046 AQ01047	CVF POND 6-S	< 1.8	81.0
AQ01047 AQ01048	CVF POND 6-3	< 1.8	77.0
AQ01048 AQ01049	CVF POND 7-1	< 1.8	99.0
AQ01049 AQ01050	CVF POND7-2	< 1.8	94.0
AQ01050 AQ01051	POND 5-1	< 1.8	89.0
AQ01051 AQ01052	POND 5-1		86.0
AQ01052 AQ01053	CVF 12-6-9	< 1.8	
AQ01053 AQ01054	CVF 12-0-9 CVF 14-3-6	< 1.8	87.0 111.0
AQ01054 AQ01055	CVF 14-3-6 CVF 14-12	< 1.8 < 1.8	113.0
AQ01055 AQ01056	CVF 14-12 CVF 15-3	< 1.8	113.0
AQ01056 AQ01057	CVF 15-3 CVF 16-3-6	30.0	114.0
AQ01057 AQ01058	CVF 10-3-0	< 1.8	105.0
AQ01058 AQ01059	CVF 16-3 CVF P-2-1	< 1.8	107.0
AQ01039 AQ01060	CVF P2-2	< 1.8	104.0
AQ01060 AQ01061	CVF P2-3	< 1.8	103.0
AQ01061 AQ01062	CVF P2-3 CVF 18-3-6	8.9	103.0
AQ01062 AQ01063	CVF 18-3-0	5.7	104.0
AQ01063 AQ01064	CVF 18-1	< 1.8	102.0
AQ01064 AQ01065	CVF 19-1	< 1.8	102.0
AQ01065 AQ01066	CVF 19-3-6	4.0	106.0
MEAN	O V F 18-3-0	4.0	92.3
SD			12.2
CV			13.2
n			36

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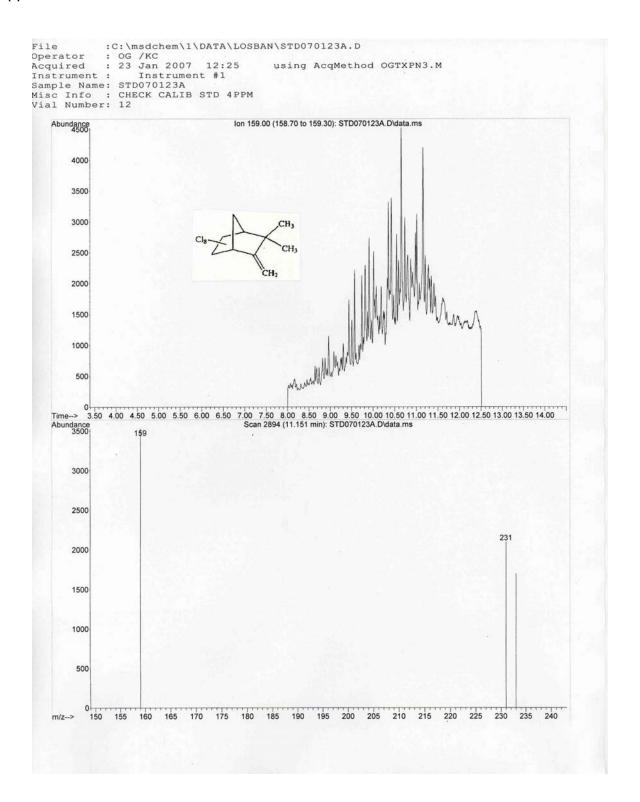
Figure 1. Site Mitigation Sampling Plan and photograph of abandoned site





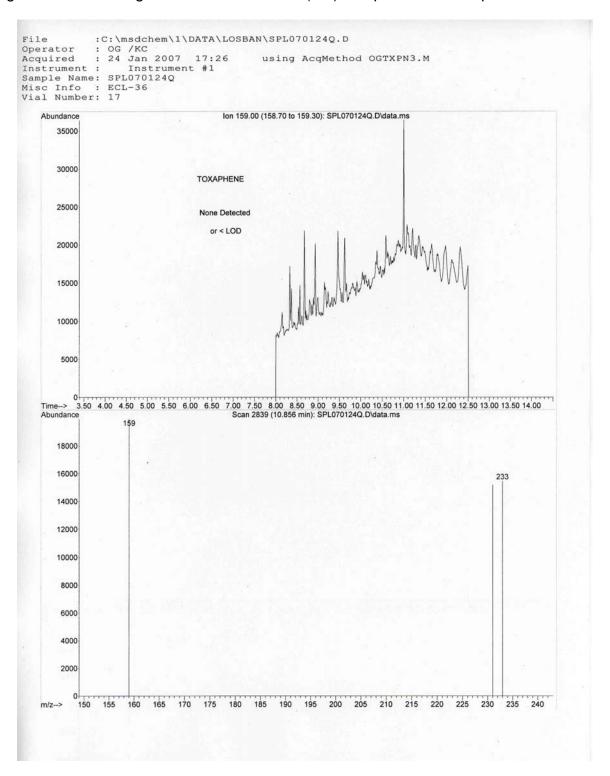
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Figure 2. Chromatogram of the extracted ion 159 m/z from toxaphene standard at 4 ppm.



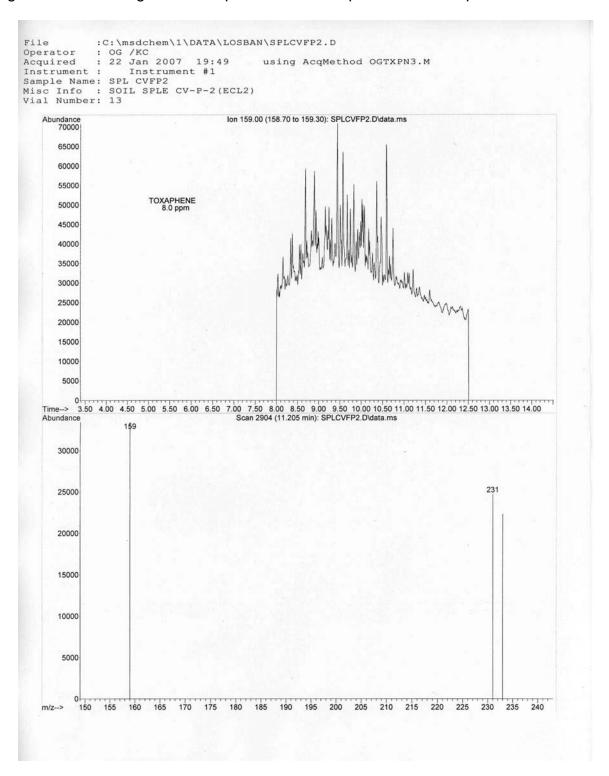
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Figure 3. Chromatogram of a non-detected (ND) toxaphene soil sample.



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Figure 4. Chromatogram of toxaphene found in a positive soil sample.



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Appendix – Sample Reports

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For ECL No.s AQ01031 to AQ01066

Auth. No. 06EC0154

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						Date Co	llected:	1/22-23/2007
equestor Address:	Maria Gill					Date Receive		1/22-23/200
	DTSC Regio					Date Ex	tracted:	1/22-24/200
	Sacramento	, CA				Data A	malyzed:	1/22-24/200
						Extraction		Hexane Extra
						Analysis	Method:	GCMSD
ampling Location:		lley Fertil	izer		Extraction	Holding Tim	e Met ?:	YES
	Azuza Rd				Analysis	Holding Tim	e Met ?:	YES
	Dos Palos,							
xtraction Methods:	EPA 3510C	For aqueous sam	ples; Separator	y funnel extrac	tion with methyl	ene chloride. So	lvent exchange	to hexane.
Actuseton mount								
		CONTROL OF STREET CONTROL	entestion	extraction with	hexans/acetone,	IOTIOMER DA POT	seur excuenda	LO MEXAME.
	EPA 3580A	For oils &	organic liq	uid samples	; Solvent di	lution with	rioned in the	report).
leanup Method:	EPA 3620	Florisil colu	mn cleanup; (N	ormally not pe	erformed unless	otherwise men	DB-5 capil	lary column
nalytical Method:	EPA 8081A	Analysis by	DB-1701 capi	llary column	GC/ECD & COL	AQ01035	Method	T
CL Number:>		AQ01031	AQ01032	AQ01033	AQ01034 BH-10-6-9	CVF-13-6-9	Blank	OL
Collector's Sample #		CVF P-1	CVF P-2	CVF P-3		soil	soil	
Sample Matrix:		soil	soil	soil	soil	mq/Kq	mg/Kg	mg/Kg
Jnits:		mg/Kg	mg/Kg	mg/Kg	mg/Kg	ilig/ kg	11197.119	
Compound	CAS #				-	ND	ND	1.0
OXAPHENE	001-35-2	ND	8.0	6.8	ND	ND	IND	
								-
								-
				-	-			
		-			1			
		-						
				20% THO	Control Ward			
Surrogate Standard Re-	covery:		1 22 2	Unit: Pero	76.0	79.0	101	
DECACHLOROBIPHENYL	051-24-3	81.0	81.0	82.0	76.0	13.0		
		3.0						

ents:		7.0	
Analyzed by:	Orlando Garbin	Signature:	3/1
Reviewed by:	Modam Gill	Modan Gill Signature	3.
Approved by:	Keh-chuh Ting	Signature:	<u> </u>

For ECL No.s AQ01031 California Department of Toxic Substances Control Environmental Chemistry Laboratory
700 Heinz Avenue Suite 100, Berkeley, CA 94710, Ph (510) 540-3003 06EC0154 Laboratory Report For...Toxaphene of Date Collected: 1/22-23/2007
Date Received by Lab: 1/22-21/2007 Maria Gillette Requestor Address: DTSC Region I Date Extracted: 1/22-24/2007
Data Analyzed: 1/22-24/2007 Extraction Method: Analysis Method: GCMSD Central Valley Fertilizer Sampling Location: Extraction Holding Time Met ?: YES Azuza Rd Analysis Holding Time Met ?: Dos Palos, CA EPA 3510C Por aqueous samples; Separatory funnel extraction with methylene chloride. Solvent exchange to hexane Extraction Methods: EPA 3546 For solid samples; microwave extraction with hexane/acctone, followed by solvent exchange to hexane. EPA 3550B For solid samples; sonication extraction with hexane/acatons, followed by solvent exchange to hexane.

EPA 3580A For oils & organic liquid samples; Solvent dilution with hexane. EPA 3620 Florisil column cleanup; (Normally not performed unless otherwise mentioned in the report). Cleanup Method: EPA 8081A Analysis by DB-1701 capillary column GC/ECD & confirmation by DB-5 capillary column Analytical Method AQ01036 AQ01037 AQ01038 AQ01039 AQ01040 Method ECL Number: ---> POND 2-S POND 3-24" Blank POND 2-2 POND 1-24 POND 2-12 Collector's Sample # soil soil soil soil soil soil Sample Matrix: mg/Kg mg/Kg mg/Kg mg/Kg mg/Kg mg/Kg Units: Compound CAS # ND ND ND ND ND 001-35-2 ND TOXAPHENE Surrogate Standard Recovery: 88.0 88.0 87.0 DECACHLOROBIPHENYL 051-24-3 85.0 QL = Quantitation Limit = Lowest calibration standard x dilution factor ND = Not detected or detected but below QL $\,$ Comments: Orlando Garbin Analyzed by: Modam Gill Reviewed by: Approved by: Keh-chuh Ting

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For ECL No.s AQ01031 to AQ01066

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		Laboratory					Auth. No.	06EC0154
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Requestor Address:	Maria Gil	lette				Date (Collected:	1/22-23/200
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	Sacramento, CA Date Extracted: 1/							
	7.						Analyzed:	1/22-24/200
						Extraction		Hexane Extr
						Analysis	s Method:	GCMSD
Sampling Location:		alley Ferti	lizer					
	Azuza Rd		-			Holding Ti		YES
	Dos Palos	, CA	5		Analysis	Holding Ti	me Met ?:	YES
Extraction Methods: Cleanup Method: Analytical Method:	EPA 3550B EPA 3550B EPA 3580A EPA 3620	For solid samp For solid samp For oils & Florisil colu	mples; Separator ples; microwave e ples; sonication organic liq umn cleanup; (N DB-1701 capi	extraction with a extraction with uid samples; formally not pe	hexane/acetone, hexane/acetone, ; Solvent di erformed unless	followed by sol followed by so lution with otherwise mer	event exchange to olvent exchange of hexane. ntioned in the	o hexane. to hexane. report).
ECL Number:>	DIN OUUIN	A0001041	A001042	A001043	AQ01044	A001045	Method	ary column
Collector's Sample #		POND 4-5	POND 4-12	POND 4-2	POND 5-S	CVF POND 6-1	Blank	OL
Sample Matrix:		soil	soil	soil	soil	soil	soil	Qu
Units:		mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mq/Kq	mg/Kg
Compound	CAS #	3, 3	2		34,53	200	11136.113	7.37.19
TOXAPHENE	001-35-2	ND	ND	ND	ND	ND	ND	1.0
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								1100
A								

QL = Quantitation Limit = Lowest calibration standard x dilution factor ND = Not detected or detected below QL

nts:			
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Analyzed by:	Orlando Garbin	Signature:	3/0 Date
Reviewed by:	Modam Gill	Modan Gill Signature:	3_7
Approved by:	Keh-chuh Ting	Signature:	3.7

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equestor Address:	Maria Gill					Date Receiv	ed by Lab:	1/22-23/200
	DTSC Regio					Date Ex	xtracted:	1/22-24/200
	Sacramento	, CA				Data	Analyzed:	1/22-24/200
						Extraction		Hexane Extr
				- 2				
						Analysis	Method:	GCMSD
ampling Location:	Central Va	lley Ferti	lizer					1000000
	Azuza Rd				Extraction	Holding Tim	ne Met ?:	YES
	Dos Palos,	CA			Analysis	Holding Tim	ne Met ?:	YES
xtraction Methods:	EPA 3510C	For aqueous sar	mples: Separator	y funnel extrac	tion with methyle	ne chloride. So	olvent exchange	e to hexane.
ACTACCION MCCHOGE	PD3 3546	Por solid same	les: microwave e	etraction with 1	nexane/acetone, !	followed by solv	rent exchange	co nexane.
	DDA SECOD	Pow solid samp	les conication	extraction with	hexane/acetone.	followed by sol	vent exchange	to hexane.
	EPA 3550B	For soils f	erania lia	wid camples	; Solvent di	lution with	hexane.	
	EPA 3580A	FOI OILS W	organic iiq	uru Sampres	rformed unless	otherwise men	tioned in the	report).
leanup Method:	EPA 3620	Florisil colu	mn cleanup; (N	ormally not pe	riormed unless	Ei	DD C comil	lami colum
nalytical Method:	EPA 8081A	Analysis by	DB-1701 capi	llary column	GC/ECD & con	firmation by	DB-5 Capit	Tary cordina
CL Number:>	- '	AQ001046	AQ01047	AQ01048	AQ01049	AQ01050	Method	
ollector's Sample #		CVF POND 6-2	CVF POND 6-S	CVF POND 7-1	CVF POND 7-2	CVF POND 7-S	Blank	QL
		soil	soil	soil	soil	soil	soil	
ample Matrix:					mg/Kg	mg/Kg	mg/Kg	mg/Kg
nits:		mg/Kg	mg/Kg	mg/Kg	mg/kg	1119/129	mg/ ng	
ompound	CAS #	Anna and an and						-
OXAPHENE	001-35-2	ND	ND	ND	- ND	ND	ND	1.0
VARRENE								
	-		-	120000				0.01
								-
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								-
							1.10.75	
		-+-						
								-
							-	
				-				
				590 90	1000000			
Surrogate Standard Rec	covery:			Unit: Perce	ent (%)			
		78.0	81.0	Unit: Perce	ent (%)	94.0	101	
	covery:	78.0	81.0			94.0	101	
Surrogate Standard Rec DECACHLOROBIPHENYL		78.0	81.0			94.0	101	
DECACHLOROBIPHENYL	051-24-3			77.0	99.0		101	
DECACHLOROBIPHENYL Note: QL = Quantite	051-24-3	Lowest ca	libration st	77.0	99.0		101	
DECACHLOROBIPHENYL Note: QL = Quantite	051-24-3	Lowest ca	libration st	77.0	99.0		101	
Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
OCCACHLOROBIPHENYL Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
DECACHLOROBIPHENYL Note: QL = Quantite	051-24-3	Lowest ca	libration st	77.0	99.0		101	
OCCACHLOROBIPHENYL Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0		101	
DECACHLOROBIPHENYL Note: QL = Quantite ND = Not Comments:	ation Limit -	Lowest ca	libration st	77.0	99.0	r	101	3/07
Note: QL = Quantite ND = Not	051-24-3	Lowest ca	libration st	77.0	99.0	r	101	3/07/
DECACHLOROBIPHENYL Note: QL = Quantite ND = Not Comments:	ation Limit -	Lowest ca	libration st	77.0	99.0	r	101	3/07/ Date:
DECACHLOROBIPHENYL Note: QL = Quantite ND = Not Comments:	ation Limit -	Lowest ca	libration st	77.0	99.0	r	101	3/07/ Date:
DECACHLOROBIPHENYL Note: QL = Quantite ND = Not Comments:	ation Limit -	Lowest ca.	libration st	77.0	99.0	r	101	3/07/ Date:

Approved by:

Keh-chuh Ting

For ECL No.s AQ01031 to AQ01066

Auth. No. 06EC0154

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								8
						Date Co	llected:	1/22-23/200
Advage.	Maria Gill	ette				Date Receive	d by Lab:	1/22-23/20
questor Address:	DTSC Regio	DTSC Region I				Date Ex	tracted:	1/22-24/20
	Sacramento	cramento, CA				Data A	nalyzed:	1/22-24/200
						Extraction		Hexane Ext
						Analysis	Method:	GCMSD
ampling Location:	Central Va	lley Fertil	izer		Extraction	Holding Tim	e Met ?:	YES
mapring Document	Azuza Rd Dos Palos,	CA			Analysis	Holding Tim	e Met /:	YES
			-les Cenaratori	funnel extract	ion with methyle	ene chloride. So	lvent exchang	e to hexane.
ktraction Methods:	EPA 3510C	For aqueous sam	ples; Separator;	straction with h	ion with methyle exame/acetone, i hexame/acetone,	followed by solv	ent exchange	to nexane.
	EPA 3546	For solid sampl	es; micions:	extraction with	hexane/acetone, thexane/acetone, solvent di	followed by sol	vent exchange	to nexame.
	EPA 3550B	For solid sampa	organic lig	uid samples	Solvent di	lution with	nexane.	e report).
	EPA 3580A	Plorieil colu	mn cleanup; (N	ormally not pe	Solvent di rformed unless GC/ECD & con	otherwise men	noned in Ci	lary colum
leanup Method:	EPA 3620	analysis by	DB-1701 capi	llary column		firmation by	UB-3 Capi	1
nalytical Method:	EPA SUSIA	AQ001051	AQ01052	AQ01053	110.00	CONTRACTOR OF COLUMN	Blank	OL
CL Number:>		POND 5-1	POND 5-2	CVF 12-6-9			The state of the s	Va
ollector's Sample #		soil	soil	soil	soil	soil	soil	mg/Kg
ample Matrix:		mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/ Mg
nits:	1 2 4	mg/kg						1.0
Compound	CAS #	ND	ND	ND	ND	ND	ND	1.0
OXAPHENE	001-35-2	ND						
							-	
								A Value
						-	ALC: NO	
					-			
				-	100			
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				1				
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			-					
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		1						-
		-		5			-	
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						1		
	_					-		
						-		
			No. of the last of			77-11-11	2	
				Unit: Per	cent (%)	7 415	101	
Surrogate Standard I	Recovery:	3 89.0	86.0	87.0	111	113	101	-
DECACHLOROBIPHENYL	051-24-	3 05.0						
D D O C								

mments:			
		011	3/07
Analyzed by:	Orlando Garbin	Signature:	7 Date 7.7
Reviewed by:	Modam Gill	Modam Crill Signature:	Date
Approved by:	Keh-chuh Ting	Signature:	Date

For ECL No.s AQ01031 to AQ01066 Auth. No. 06EC0154

							Page	0	
							of	8	
Requestor Address:	Maria Gill	ette					ollected:	1/22-23/2007	
requestor Address.	DTSC Regio					Date Receiv		1/22-23/2007	
	Sacramento	, CA					xtracted:	1/22-24/2007	
	-					Extraction	Analyzed:	Hexane Extrn	
						Analysis		GCMSD	
			Part Francisco			Allalysis	raccinou i		
Sampling Location:		lley Ferti	lizer		Pytraction	Holding Tir	ne Met ?:	YES	
	Azuza Rd	77				Holding Tir		YES	
	Dos Palos,								
Extraction Methods:	EPA 3510C For aqueous samples, Separatory funnel extraction with methylene chloride. Solvent exchange to her EPA 3546 For solid samples, microwave extraction with hexane/acetone, followed by solvent exchange to hexat EPA 3550B For solid samples; sonication extraction with hexane/acetone, followed by solvent exchange to hexat EPA 3580A For oils & organic liquid samples; Solvent dilution with hexane. EPA 3620 Floristi column cleanup; (Normally not performed unless otherwise mentioned in the report EPA 8081A Analysis by DB-1701 capillary column GC/ECD & confirmation by DB-5 capillary column and the second s								
Analytical Method:	EPA 8081A			liary column	A001059	A00160	Method	Y	
ECL Number:>		AQ001056	AQ01057	AQ01058	CVF P-2-1	CVF P2-2	Blank	QL	
Collector's Sample #		CVF 15-3	CVF 16-3-6	CVF 18-S	soil	soil	soil		
Sample Matrix:		soil	soil	soil	mq/Kq	mg/Kg	mg/Kg	mg/Kg	
Units:		mg/Kg	mg/Kg	mg/Kg	mg/ kg	mg/ mg			
Compound	CAS #			4000	ND	ND	ND	1.0	
TOXAPHENE	001-35-2	ND	30*	ND	ND	ND	115		
Surrogate Standard Re				Unit: Perc	ent (%)	104	101		
DECACHLOROBIPHENYL	051-24-3	113	114	105	107	104	101		
THE RESERVE OF THE PERSON NAMED IN COLUMN TWO IS NOT THE PERSON NAMED IN COLUMN TWO IS NAMED IN COLUMN TW									

ND = Not detected or detected below QL

Comments: * Detected value exce	eded linear calibration. T	his is an estimate.	
Analyzed by:	Orlando Garbin	Sarfury Signature:	3/07/0,
Reviewed by:	Modam Gill	Meelan Gill Signature	3-7.07 Date:
Approved by:	Keh-chuh Ting	K. C. Ting	3.7.07

700 Heinz Av Laboratory Gillette Region I mento, CA al Valley Pert: Rd alos, CA	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	y funnel extract ktraction with extraction wit	Extraction Analysis tion with methyl hexame/acetone. hexame/acetone.	Date (Date Recei Date I Data I Extraction Analysis Holding Ti Holding Ti ene chloride S followed by sol	me Met ?: me Met ?: me Met ?:	06EC0154 7 8 1/22-21/200 1/22-24/200 1/22-24/200 Hexane Extri GCMSD YES YES								
Gillette Region I mento, CA al Valley Pert: Rd alos, CA 510C For aqueous sa 550B For solid samp 550B For solid samp 580A For oils & 20 Florisit col 201A Analysis by AQ001061 CVF P2-3	ilizer ilizer ilizer insples, Separator, Separator, Separator, Separator, Separator, Separator, 1701 capi. AQ01062	y funnel extrac ktraction with extraction with extraction with id samples promally not	Extraction Analysis tion with methyl hexame/acetone. hexame/acetone.	Date (Date Recei Date I Data I Extraction Analysis Holding Ti Holding Ti ene chloride S followed by sol	Page of Collected: ved by Lab: Extracted: Analyzed: Method: Method: me Met ?: me Met ?:	7 8 1/22-23/200 1/22-23/200 1/22-24/200 1/22-24/200 HEXARE EXET GOMED YES YES								
Region I mento, CA al Valley Pert: Rd alos, CA 510C For aqueous st 546 For solid samp 580A For oils & 520 Florisit colu 081A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Date Recei Date I Date I Data Extraction Analysis Holding Ti Holding Ti ene chloride. S followed by sol	Page of Collected: ved by Lab: Extracted: Analyzed: Method: Method: me Met ?: me Met ?:	7 8 1/22-23/200 1/22-23/200 1/22-24/200 1/22-24/200 HEXARE EXTR GCMSD YES YES								
Region I mento, CA al Valley Pert: Rd alos, CA 510C For aqueous st 546 For solid samp 580A For oils & 520 Florisit colu 081A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Date Recei Date I Date I Data Extraction Analysis Holding Ti Holding Ti ene chloride. S followed by sol	of Collected: ved by Lab: Extracted: Analyzed: 1 Method: 5 Method: me Met ?: me Met ?:	8 1/22-23/200 1/22-23/200 1/22-24/200 1/22-24/200 Hexane Extr GCMSD YES YES								
Region I mento, CA al Valley Pert: Rd alos, CA 510C For aqueous st 546 For solid samp 580A For oils & 520 Florisit colu 081A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Date Recei Date I Date I Data Extraction Analysis Holding Ti Holding Ti ene chloride. S followed by sol	ved by Lab: Extracted: Analyzed: 1 Method: 5 Method: me Met ?: me Met ?:	1/22-21/200 1/22-24/200 1/22-24/200 Haxane Extr GOMSD YES YES								
Region I mento, CA al Valley Pert: Rd alos, CA 510C For aqueous st 546 For solid samp 580A For oils & 520 Florisit colu 081A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Date Recei Date I Date I Data Extraction Analysis Holding Ti Holding Ti ene chloride. S followed by sol	ved by Lab: Extracted: Analyzed: 1 Method: 5 Method: me Met ?: me Met ?:	1/22-21/200 1/22-24/200 1/22-24/200 Haxane Extr GOMSD YES YES								
al Valley Pert: Rd alos, CA 510C For aqueous se 5466 For solid samp 550B For solid samp 580A For oils & 520 Florisi colu 81A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Date I Data Extraction Analysis Holding Ti Holding Ti ene chloride. S	Extracted: Analyzed: Method: Method: Method: me Met ?: me Met ?:	1/22-24/200 1/22-24/200 Haxane Extr GCMSD YES YES								
Rd alos, CA 510C For squeous as 546 For solid samp 580A For oils & 520 Florisit colu 881A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Extraction Analysis Holding Ti Holding Ti ene chloride. S followed by sol	Method: Method: me Met ?: me Met ?:	1/22-24/200 Hexane Extr GCMSD YES YES								
Rd alos, CA 510C For squeous as 546 For solid samp 580A For oils & 620 Florisi colo 81A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Analysis Holding Ti Holding Ti ene chloride. S followed by sol	me Met ?: me Met ?: me Met ?:	YES YES								
Rd alos, CA 510C For squeous as 546 For solid samp 580A For oils & 620 Florisi colo 81A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Holding Ti Holding Ti ene chloride. S	me Met ?: me Met ?:	YES YES								
Rd alos, CA 510C For squeous as 546 For solid samp 580A For oils & 620 Florisi colo 81A Analysis by AQ001061 CVF P2-3	imples; Separator; les; microwave e: cles; sonication organic liq umn cleanup; (No DB-1701 capi:	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Holding Ti ene chloride. S followed by sol	me Met ?:	YES								
alos, CA 510C For squeous ss 546 For solid samp 550B For solid samp 580A For oils & 520 Florisil col 081A Analysis by AQ001061 CVF P2-3	oles; microwave es oles; sonication organic liquumn cleanup; (No DB-1701 capi	straction with extraction with uid samples ormally not pe	Analysis tion with methyl hexane/acetone. hexane/acetone, y Solvent di	Holding Ti ene chloride. S followed by sol	me Met ?:	YES								
546 For solid samp 550B For molid samp 580A For oils & 520 Florisil column 81A Analysis by AQ001061 CVF P2-3	oles; microwave es oles; sonication organic liquumn cleanup; (No DB-1701 capi	straction with extraction with uid samples ormally not pe	hexane/acetone, hexane/acetone, ; Solvent di	followed by sol	Colvent exchange	-								
546 For solid samp 550B For molid samp 580A For oils & 520 Florisil column 81A Analysis by AQ001061 CVF P2-3	oles; microwave es oles; sonication organic liquumn cleanup; (No DB-1701 capi	straction with extraction with uid samples ormally not pe	hexane/acetone, hexane/acetone, ; Solvent di	followed by sol	colvent exchange									
550B For solid samp 580A For oils & 520 Florisil col 081A Analysis by AQ001061 CVF P2-3	organic liquumn cleanup; (No DB-1701 capi	extraction with uid samples ormally not pe	hexane/acetone, Solvent di	followed by sol		EPA 3510C For aqueous samples; Separatory funnel extraction with methylene chloride. Solvent exchange to hexane.								
580A For oils & 520 Florisil colo 081A Analysis by AQ001061 CVF P2-3	organic liquumn cleanup; (No DB-1701 capi	uid samples ormally not pe	: Solvent di		vent exchange	EPA 3546 For solid samples; microwave extraction with hexane/acetone, followed by solvent exchange to hexane. EPA 3550B For solid samples; sonication extraction with hexane/acetone, followed by solvent exchange to hexane.								
520 Florisil cole 081A Analysis by AQ001061 CVF P2-3	DB-1701 capi AQ01062	ormally not pe	, gozyene dr	lution with	hevene	to hexane.								
AQ001061 CVF P2-3	DB-1701 capi	llary column	erformed unless	otherwise mer	tioned in the	report)								
AQ001061 CVF P2-3	AQ01062		GC/ECD & con	firmation by	DB-5 capil	ary column								
	OTTO TO T	AQ01063	AQ01064	AQ01065	Method									
soil	CVF 18-3-6	CVF 18-1	CVF 19-1	CVF 19-S	Blank	Or								
	soil	soil	soil	soil	soil									
mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg								
		100 0 300		MARKET 1872	naonaeo	2 (5)(3)(5)								
5-2 ND	8.9	5.7	ND	4.0	ND	1.0								
-														
				-		-								
		nie Dans	a 6 10.5		1									
-2 102				100	- V V									
2 103	103	104	102	102	101									
	1-3 103			Unit: Percent (%) 1-3 103 104 102										

Signature: Date:

Reviewed by:

Approved by:

Modam Gill

Keh-chuh Ting

For ECL No.s AQ01031 California Department of Toxic Substances Control Environmental Chemistry Laboratory 700 Heinz Avenue Suite 100, Berkeley, CA 94710, Ph (510) 540-3003 to AQ01083 06EC0154 Laboratory Report For... Toxaphene Date Collected: 1/22-21/2007

Date Received by Lab: 1/22-23/2007

Date Extracted: 1/22-24/2007

Data Analyzed: 1/22-24/2007

Extraction Method: Hexame Extra Maria Gillette Requestor Address: DTSC Region I Sacramento, CA Extraction Method: Analysis Method: GCMSD Central Valley Fertilizer Sampling Location: Extraction Holding Time Met ?: YES
Analysis Holding Time Met ?: YES Dos Palos, CA EPA 3510C For aqueous samples; separatory funnel extraction with methylene chloride. Solvent exchange to hexane.

EPA 3546 For solid samples; microwave extraction with hexane/acetone, followed by solvent exchange to hexane.

EPA 3550B For solid samples; sonication extraction with hexane/acetone, followed by solvent exchange to hexane.

EPA 3580A For oils & organic liquid samples; Solvent dilution with hexane.

EPA 3620 Florist column cleanup; (Normally not performed unless otherwise mentioned in the report). Extraction Methods: Cleanup Method: EPA 8081A Analysis by DB-1701 capillary column GC/ECD & confirmation by DB-5 capillary column Analytical Method: Method AQ01066 ECL Number: ---> Blank QL CVF 19-3-6 Collector's Sample # soil Sample Matrix: soil mg/Kg mg/Kg mg/Kg Units: CAS # Compound ND 1.0 001-35-2 TOXAPHENE Unit: Percent (%) Surrogate Standard Recovery: 051-24-3 106 DECACHLOROBIPHENYL Note: QL = Quantitation Limit = Lowest calibration standard x dilution factor ND = Not detected or detected below QL Comments: Analyzed by: Orlando Garbin Modam Gill Reviewed by: Keh-chuh Ting Approved by: